The reaction flask was then maintained at 100ºC for a total of 6 hours then cooled down to ambient laboratory temperature. Next the contents of the flask were transferred into 2 litres of methanol containing 10% de-ionised water in an ice-cooled 5 litre beaker. Immediately an off-white solid precipitate came out of solution, this was filtered off and collected. The crude solid was recrystallised a total of 4 times from a tetrahydrofuran/methanol solution producing a white solid product.

The product was obtained in a quantity of 31.5g which was a 37% yield. It had a melting point of 110ºC.

Step 2, partial de-acylation
Glacial acetic acid (2.04g) was added slowly dropwise with stirring into a solution of ethylenediamine (4.09g) in tetrahydrofuran (THF, 850cm^3). A white precipitate formed which remained during the reaction. α-Cellobiose octanonanoate (50g) was then added and the whole reaction mixture stirred at room temperature for a total of 48 hours.

At the end of the reaction period, the contents of the flask were transferred to a two litre separating funnel, 350cm^3 of water was added and the mixture extracted with dichloromethane (250cm^3). The organic layer was collected and further washed with successive 350cm^3 portions of (1) dilute HCl (0.1M), (2) aqueous sodium bicarbonate (1M) and (3) water.

The resultant organic phase was recovered, dried over anhydrous magnesium sulphate, filtered and the remaining solvent removed by rotary evaporation. A slightly sticky off-white crude solid was obtained. This was then re-crystallised from a mixture of THF/methanol (50:300cm^3). During overnight storage, a white solid precipitated out and was filtered off, dried and collected, yielding 30.5g of a white free-flowing solid as intermediate product (68% Yield).

Step 3.

3A - Re-acylation with an acyl chloride

This route is exemplified for the benzoate ester, and is useable for all the esters by substituting the other acid chlorides for benzoyl chloride.

A 3 neck 500cm^3 round bottomed flask was charged with cellobiose heptanonanoate (5g, $3.78x10^{-3}$moles) together with 125cm^3 of toluene. The mixture was stirred thoroughly until a clear solution resulted. Next triethylamine (0.479g, $4.73x10^{-3}$moles) was slowly added dropwise to the solution.

Thereafter, Benzoyl Chloride (0.665g, $4.73x10^{-3}$moles) was added slowly and cautiously via a pressure equalising dropping funnel into the reaction mix. When addition of the reagents was complete, the whole reaction solution was heated up to and maintained under reflux conditions for a total of 2-3hrs. The flask was then removed from the heat and after cooling was filtered to remove the solid triethylamine hydrochloride salt present. A clear straw

coloured liquid was obtained. All solvent was then removed by rotary evaporation to give a crude product, a straw coloured gel-like material. The crude product was re-crystallised from THF-MeOH ($20cm^3$:$120cm^3$). The resultant product, a white free-flowing solid, was filtered off, collected and dried at 40-45°C. Yield was 3.5g (65%)

3B Re-acylation employing an acid/catalyst

This method is exemplified using benzoic acid and can also be used for making the other cellobiose esters by replacing benzoic acid by the appropriate acid.

A 2 neck $250cm^3$ round bottomed flask was charged with Benzoic Acid (29.54g, 0.24moles) and trifluoroacetic anhydride(19.05g, 0.091moles). The mixture was stirred and heated to and maintained at 100°C for one hour. Cellobiose heptanonanoate (5g, $3.78x10^{-3}$moles) was introduced slowly via a solids addition funnel into the activated solution. After it had added completely, the reaction mixture was maintained at 100°C stirred for a total of 6 hours. The reaction flask was then cooled down to room temperature. An ice-cooled solution of methanol-water ($400cm^3$ MeOH:$40cm^3$ water) was poured into the flask, whereupon a solid precipitate formed immediately, was filtered off and re-crystallised from THF-MeOH ($20cm^3$:$120cm^3$). The resultant product was filtered off collected and re-crystallised a second time from THF-MeOH to remove trace acid. The final product, a white solid, was filtered off, collected and dried at 40-45°C. The yield was 3.1g (58%)